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**Northwestern University**  
**2802 Maple Avenue**  
**Evanston, Illinois 60201-3135**

**Final Report**

**GRANT NUMBER F49620-95-1-0501**

**TO**

**UNITED STATES AIR FORCE**  
**AIR FORCE OFFICE OF SCIENTIFIC RESEARCH**

**FOR**

**A NANO-MODULATED CERAMIC COATING**  
**DEPOSITION SYSTEM**

by

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## **1. INTRODUCTION**

This is the final report on A Nano-Modulated Ceramic Coating Deposition System. This effort was funded by the United States Air Force Office of Scientific Research with a grant of \$350,000 and a BIRL/Northwestern University contribution of \$ 122,128, for a total of \$472,128. The funding grant provided for the design and construction of a sputter deposition system capable of producing nano-layer composites of ceramic materials. The work on this system was started in August 1995. Delivery of the deposition chamber was in February 1996. Peripheral equipment, such as pumps, power supplies, and gauging was delivered such that the equipment would be available when needed.

The site for the system was prepared prior to the delivery of the chamber. Sight preparation consisted of providing sufficient water, electrical power, and air to operate the system. Delivery of the chamber was the first step in the construction of the deposition system. The various purchased items were installed onto the chamber and checked for functionality. This included the pumps for the chamber and load lock, the sputter sources, the power supplies and all of the associated measuring equipment.

The deposition system is now fully functional. Base pressures in the load lock and the deposition chamber itself are in the  $10^{-7}$  Torr range. The chamber is fully instrumented, with the exception of the ellipsometer. The ellipsometer is still being used on the bench to measure coating optical properties after deposition. The ellipsometer will be installed and used as a process control once we are thoroughly familiar with its operation and capabilities.

Several graduate students from the Materials Science Department at Northwestern University are currently sharing the use of the deposition chamber for experimental work on their theses. This work is being supported by both Air Force (AFOSR) and industrial contracts.

### **1.1. Equipment Description**

The deposition system is shown schematically in Figure 1. To avoid clutter, we have not included all of the peripheral equipment in the schematic diagram. The system is equipped with cryogenic high vacuum pumps on both the main chamber and the load lock. Both the main chamber and the load lock have base pressures in the low  $10^{-7}$  Torr range. The load lock will achieve a vacuum level of approximately  $1 \times 10^{-5}$  from atmosphere within 5 minutes after initiating the pumping cycle. At this pressure level in the load lock, very little contamination is transferred into the deposition chamber. This means that the deposition chamber recovery time (the time required to achieve the desired base pressure after exposure to contamination) is very short, typically on the order of a few minutes. The single roughing pump on the system serves both to rough out the load lock and to rough out the deposition chamber when it is vented to atmosphere for target changes.

The sputtering sources are strongly unbalanced magnetrons arranged in a closed field configuration. This set up produces a maximum in ionization at the substrate. The cathodes are mounted through the doors of the deposition system, on umbilicals which can be adjusted to change the target to substrate distance. The cathodes are capable of accepting either

directly cooled targets or targets mounted to backing plates. This allows the use of a wider variety of target materials other than just metals, i. e., hot pressed materials can also be sputtered.

A substrate table has been designed and built. The table has a variable speed rotation capability and can be biased with either rf, dc or pulsed dc power. Designed into the table, but not yet built into it, are capabilities for thermocouple and Langmuir probe measurements. These capabilities will be added as required for the various programs that the deposition system will be used for. Also designed into the substrate table, but not yet implemented, is the capability to heat to temperatures suitable for epitaxial growth of some thin film materials.

Power to the cathodes is provided by 10 kW Advanced Energy power supplies equipped with SPARC-LE units which are capable of operation at frequencies up to 100 kHz. The frequency range is important because higher frequencies will prevent arcing from oxides, or other insulating materials, growing on the target surface. A 10 kW Advanced Energy power supply with a SPARC-LE unit has also been provided for substrate bias. The variable frequency SPARC-LE units can be synchronized together to minimize problems with plasma shutdowns when different frequencies or duty cycles are used. Presently, up to three SPARC-LE units can be synchronized together.

A bias frequency higher than that which can be supplied by the variable frequency SPARC-LE units will occasionally be required. We have therefore installed a 13.56 MHz rf bias capability at the substrate. We have also included in the design of the system an rf probe that will measure the real voltage, current, and impedance of the plasma near the substrate surface. Knowledge of the true values of these parameters will greatly assist in developing processes which require an rf bias.

The deposition system has been designed with the latest and best process controls available to us at the time of construction. We have installed a Spinning Rotor Gauge to measure true chamber pressure, a STABIL™ ion gauge which, because it doesn't drift, will give an accurate chamber pressure measurement over a long period of time, a high accuracy capacitance manometer for measurement of true deposition pressures, a fast response mass spectrometer for control of the reactive gas partial pressure, and a modified gas flow control system with quick response piezoelectric valves to rapidly change the gas flow into the system as required by the deposition process.

A variable wavelength ellipsometer has been purchased to install on the deposition chamber as part of a control loop for control of coating properties. For in-situ use, this instrument will be set up to monitor only a single wavelength rather than the entire visible spectrum. This will insure that the response time of the instrument is such that corrections to relevant deposition parameters can be made in order to keep the deposition process on track. Currently, this instrument is being used on the bench to characterize the optical properties of the coatings being deposited.

Substrate heat has been provided for, but not yet installed into the deposition chamber. Heat will be installed when required by the coating work being conducted in the chamber. The heater should be capable of temperatures up to approximately 400 degrees Celsius.

A computer has been designated for use with the deposition system. Hardware for deposition process control has been purchased, and we are currently writing the software required for its use. When complete, the system will be capable of operation either manually or under computer control.

## **1.2. Equipment purchases**

The various items of equipment, purchased with AFOSR funds, to construct this deposition system are listed in Table 1, along with their original estimated costs. A list of purchase orders issued for this project and their costs is shown in Table 2. Deviations from the originally planned purchases are discussed in the following section of this report. Some deviations were required because the design of the system had not been finalized at the time of the award of the grant, or new equipment had become available that wasn't on the market at the time the proposal was submitted. The final design was part of the Northwestern contribution to the overall program.

Table 3 lists BIRL's originally planned contributions to the overall effort. Table 4 lists AFOSR's and BIRL's contributions to the labor required to construct the deposition system from the purchased parts. The Air Force's contributions were limited to the \$ 350,000 appropriation, while BIRL's contributions exceeded the estimated \$122,128 by a significant amount.

**Table 1. Equipment purchased with AFOSR funds for construction of the Nano-Modulated Ceramic Sputter Deposition System.**

| Original Plan               |                        |                  | Actual Purchases                                |
|-----------------------------|------------------------|------------------|---|
| Item                        | Original Vendor        | Estimated Cost k | Vendor <sup>1</sup>                             |
| Deposition Chamber          | Meyer Tool             | 37               | Meyer Tool                                      |
| Load Lock                   | "                      | 12               | "   |
| Cryopump                    | CTI                    | 8                | CTI   |
| Sputter Power Supplies      | Advanced Energy        | 40               | Advanced Energy                                 |
| SPARC-LE Units              | Advanced Energy        | 12               | Advanced Energy                                 |
| RF Plasma Probe             | "                      | 7                | "   |
| Sputter Sources             | Materials Science Inc. | 20               | Genco Ltd.                                      |
| Mass Spectro-meter          | MKS Instruments        | 20               | Spectra   |
| Gas Flow Control            | MKS Instruments        | 15               | MKS Instruments                                 |
| Roughing Line Hardware      | Various                | 6.0              | Various-MKS<br>Various -MDC<br>Various - Lesker |
| Throttle Valve              | "                      | 5                | MKS Instruments                                 |
| Mechanical Pump             | Leybold-Heraeus        | 4                | Alcatel   |
| STABIL-ion Gauge            | Granville Phillips     | 4                | Granville Phillips                              |
| Gate Valves                 | Midwest Vacuum         | 12               | Midwest Vacuum                                  |
| Feed-throughs               | Various                | 8                | Various <sup>2</sup> -Lesker<br>-MDC<br>-MKS    |
| Substrate Heater            | Denton Vacuum          | 5                | CHA Industries                                  |
| Computer                    | Local Vendor           | 3                | BIRL Contribution                               |
| Control Software            | National Instruments   | 2                | National Instruments                            |
| Substrate Holder Components | Various                | 3                | Various-Meyer Tool,<br>-NU machine shop         |
| Ellipsometer                | JA Woollam Co.         | 54               | JA Woollam Co.                                  |
| Miscellaneous               | Various Vendors        | 20               | Various Vendors                                 |
| Amount Requested            |                        | \$297,000        |   |
| Amount spent                |                        | \$299,000        |   |

1. Vendors full names and addresses are given in Appendix 1.

**Table 2. Purchase orders, vendors and costs for equipment purchased for construction of the ceramic coating deposition system**

| <b>PO Number</b> | <b>Vendor</b>          | <b>Description</b>          | <b>Cost \$</b> |
|------------------|------------------------|-----------------------------|----------------|
| RX001039         | Meyer Tool             | Chamber                     | 41,000         |
| RX001041         | MKS Instr.             | Flow Cont.                  | 10,457         |
| RX001043         | Spectra                | Mass Spectr.                | 18,500         |
| 1044             | Genco Ltd.             | Sputter Sources             | 19,600         |
| 1046             | Meyer Tool             | Load Lock                   | 10,900         |
| 1052             | CTI                    | Cryopump                    | 7,995          |
| 1054             | VAT                    | Gate Valve                  | 6,795          |
| 1055             | MDC Vac. Prod.         | Adapter                     | 5,451          |
| 1091             | Adv. Energy            | RF Power Supply             | 13,585         |
| 1093             | JA Woollam             | Ellipsometer                | 54,000         |
| 1111             | Adv. Energy            | SPARC-LEs                   | 12,000         |
| PD003589         | Granville Phillips     | STABIL ion gauge            | 4,100          |
| 3590             | Alcatel                | Pump                        | 3,032          |
| 3607             | MKS Instr.             | Capacitance Manometer       | 3,660          |
| 3608             | MKS                    | Throttle Valve              | 4,626          |
| 3634             | VAT                    | Gate Valve                  | 4,185          |
| 3690             | Maxtek                 | Piezo valves                | 1,290          |
| 3691             | Austin Scientific      | Cryo absorber               | 450            |
| 3692             | Copper and Brass Sales | Hard Copper roll            | 529            |
| 3696             | Austin Scientific      | Cryo fittings               | 452            |
| 3701             | E Besler               | Import fees                 | 799            |
| 3738             | MKS                    | Vacuum fittings             | 3,529          |
| 3748             | Kurt J. Lesker         | Vac. Fittings, Feedthroughs | 4,734          |
| 3751             | Nor-Cal Products       | Foreline Trap               | 8,985          |
| 3774             | CHA Industries         | Heater Pwr Supply           | 4,007          |
| 3804             | Adv. Energy            | Power Supplies              | 37,791         |
| 3815             | Meyer Tool             | Frame Modification          | 2,490          |
| 3923             | MKS                    | Spinning Rotor Gauge        | 6,500          |
| 3966             | MKS                    | Vac. Fittings               | 1,039          |
| 3967             | Newark Electronics     | Elec. Comp.                 | 240            |
| 3983             | McMaster Carr          | Misc.                       | 327            |
| 4020             | Instr. Assoc.          | Misc.                       | 425            |
| 4077             | Newark                 | Misc. Elec.                 | 1,047          |
| 4078             | Evergreen Oak          | Wire                        | 332            |
| 4162             | Ash Equip.             | Heater                      | 274            |
| 4169             | Natl. Instr.           | Computer Hdwr.              | 1,458          |
| 4338             | Ash Equip.             | Heater                      | 270            |
| 4355             | Instr. Assoc.          | Tube Fittings               | 561            |
| 4374             | Grainger               | Fittings                    | 59             |
| 4379             | McMaster Carr          | Tube Fittings               | 189            |
| 4495             | Meyer Tool             | Subst. Fixture              | 1,830          |
| 4508             | Lesker                 | Flow Switches               | 390            |

|      |                   |                |       |
|------|-------------------|----------------|-------|
| 4511 | McMaster Carr     | Tubing         | 57    |
| 4512 | Dearborn Valve    | Valves         | 374   |
| 4513 | Bradley Supply    | Tube Fittings  | 228   |
| 4515 | Instr. Assoc.     | Connectors     | 626   |
| 4551 | Bradley Supply    | Connectors     | 54    |
| 4569 | Berry Bearing     | Bearings       | 131   |
| 4570 | Stock Drive Prod. | Sprockets      | 144   |
| 4586 | Grainger          | Gear Motors    | 642   |
| 4588 | Bradley Supply    | Ball Valves    | 52    |
| 4600 | Newark            | Misc. Elec.    | 44    |
| 4608 | Instr. Assoc.     | Connectors     | 177   |
| 4667 | Dearborn Valve    | Connectors     | 38    |
| 4668 | Instr. Assoc.     | Fittings       | 19    |
| 4669 | Nor-Cal Prod      | Adapter        | 1,660 |
| 4700 | Instr. Assoc.     | Connectors     | 72    |
| 4743 | McMaster-Carr     | Tubing, Misc.  | 196   |
| 4752 | Dearborn Valve    | Misc. Fittings | 138   |
| 4753 | Instr. Assoc.     | Misc. Fittings | 110   |
| 4774 | Meyer Tool        | Flange Assy.   | 1,580 |
| 4799 | McMaster-Carr     | Al. Rod        | 93    |
| 4800 | McMaster-Carr     | Misc. Parts    | 85    |
| 4814 | Revere Electric   | Conduit        | 53    |
| 4836 | Granville Philips | Spare Parts    | 805   |

**Table 3. BIRL's Equipment Contributions**

| Original Plan            |                   | Actual<br>Vendor Cost |
|--------------------------|-------------------|-----------------------|
| Description              | Estimated Value K |                       |
| Frame and Instr. Racks   | 4                 | BIRL Provided         |
| Utilities                | 8                 | Contractors           |
| Load Lock Cryopump       | 8                 | BIRL Provided         |
| Valve Sequencers         | 5                 | BIRL Provided         |
| Process Control Panel    | 5                 | BIRL Provided         |
| Laboratory Modifications | 10                | BIRL Provided         |
| Deposition System Design | 11                | BIRL Provided         |
| Spinning Rotor Gauge     | 12                | MKS Instruments       |
| Ion Gauge (LL)           | 2                 | BIRL Provided         |
| RF Power Supply          | 10                | Advanced Energy       |
| Total Estimated          | \$75,000          |                       |

**Table 4. AFOSR and BIRL labor dollars**

| AFOSR        |          | BIRL             |          |
|--------------|----------|------------------|----------|
| Appropriated | Spent    | Initial Estimate | Spent    |
| \$53,000     | \$51,000 | \$47,145         | \$96,000 |

Special Circumstances of Acquisition: None

Changes in Original Equipment list:

Additional Items:

1. Mass Spectrometer: Purchased from Spectra rather than MKS because of better response time and better price.
2. SPARC-LE Units: Variable frequency units became available between the time the proposal was written and the grant award. The variable frequency units are more expensive than those originally allowed for. Therefore, two of the variable frequency units were purchased with AFOSR grant money and one was purchased by BIRL.
3. RF plasma probe: This device was originally scheduled to be purchased with AFOSR grant money. Instead, the vendor, Advanced Energy, has provided a probe on an evaluation basis.
4. Sputter Sources: A vendor other than the one shown in the original proposal was chosen as the supplier of the sputter sources. The sputter sources were purchased from Gencoa because of their experience with the fabrication of unbalanced magnetrons.
5. Rough Pump: Alcatel was chosen instead of Leybold-Heraeus because of price.
6. Substrate Heater: The substrate heater power supply was purchased from CHA instead of Denton. This was done because of price.
7. Computer: BIRL contributed a computer instead of purchasing on using grant funds.
8. RF Power Supply: This item was originally to be contributed by BIRL. However, it was purchased using AFOSR funds. Additional labor dollars were contributed by BIRL to compensate for the purchase.

Several additional purchases, not itemized in the proposal, were made. These were necessitated by system design changes made after the award of the grant. As examples, several adapters were required to mate the gate valves purchased to the final chamber design.

Deleted Items: There were several changes from the original list of items to be purchased or provided. However, no items were completely eliminated.

### **1.3. Summary of Research Projects**

The deposition system is quite new and to date we have had time to use it for only one research project, which is being sponsored by AFOSR. The project entitled "Synthesis and Characterization of Oxide Superlattice Coatings" and is Grant No. F49620-95-1-0177. the scope of the work being done on this project is to use high rate reactive dc magnetron sputtering to deposit superlattice films of oxide materials. These materials are expected to have enhanced mechanical properties, such as hardness and abrasion resistance, optical properties that can be controlled by controlling ratio of the materials used in a superlattice period, and are also expected to have excellent thermal barrier properties.

## Appendix

### Vendors Used

|                        |  |
|------------------------|--|
| Chamber/Load Lock      | Meyer Tool<br>4601 Southwest Highway<br>Oak Lawn, Illinois 60453<br>(708) 425-9080                                 |
| Cryopump               | CTI<br>Technical Engineering Associates<br>7920 Lakeville Blvd.<br>Lakeville, MN 55044<br>(612) 469-6500           |
| Sputter Power Supplies | Advanced Energy Industries<br>Technical Engineering Associates<br>(see above)                                      |
| SPARC-LE Units         | Advanced Energy Industries<br>Technical Engineering Associates<br>(see above)                                      |
| RF Plasma Probe        | Advanced Energy Industries<br>1625 Sharp Point Drive<br>Fort Collins, CO 80525<br>(970) 221-4670                   |
| Sputter Sources        | Genco LTD.<br>4 Wavertree Boulevard South<br>Wavertree Technology Park<br>Liverpool, L7 9PF, UK<br>44-151-252-2200 |
| Mass Spectrometer      | Spectra<br>700 A East Dunne Ave.<br>Morgan Hill, CA 95037<br>(408) 778-6060  |
| Gas Flow Control       | MKS Instruments<br>13341 Southwest Highway<br>Orland Park, IL 60462<br>(708) 923-0222                              |
| Roughing Line Hardware | 1. MKS Instruments (see above)   |

|                                   |   |
|-----------------------------------|---|
|                                   | <ol style="list-style-type: none"> <li>2. MDC Vacuum Products<br/>Advent Associates<br/>5808 N. St. Louis Ave.<br/>Chicago, IL 60659<br/>(773) 583-7979</li> <li>3. Kurt J. Lesker<br/>1515 Worthington Ave.<br/>Clairton, PA 15025<br/>1-800-245-1656</li> </ol> |
| Throttle Valve<br>Mechanical Pump | MKS Instruments (see above)<br>Alcatel<br>Midwest Vacuum<br>201 E. Ogden Ave. Suite 15-1<br>Hinsdale, IL 60521<br>(630) 323-5399  |
| STABIL Ion Gauge                  | Granville Phillips<br>5675 Arapahoe Ave.<br>Boulder, CO 80303<br>1-800-776-6543   |
| Feedthroughs                      | Kurt J. Lesker (see above)<br>MKS Instruments (see above)<br>MDC Vacuum Products (see above)  |
| Substrate Heater                  | CHA Industries<br>Byron Ellis Associates<br>650 First Ave.<br>Des Plaines, IL 60016<br>(847) 298-7626   |
| Gate Valves                       | VAT<br>Midwest Vacuum (see above)   |
| Computer                          | BIRL contribution   |
| Control Software                  | National Instruments<br>6504 Bridge Point Parkway<br>Austin, Texas 78730-5039<br>1-800-433-3844   |
| Substrate Holder Components       | <ol style="list-style-type: none"> <li>1. Meyer Tool (see above)</li> <li>2. NU machine shop</li> </ol>   |

3. Bradley Supply Co.  
PO Box 29096  
Chicago, IL 60629  
(312) 434-7400
4. Instrument Associates  
4833 W 128 St.  
Alsip, IL 60658  
(708) 597-9880
5. Grainger  
8045 River Drive  
Morton Grove, IL 60053  
(847) 965-7600

#### Ellipsometer

JA Woollam Co.  
650 J Street  
Lincoln, NE 68508  
402-477-7501

#### Miscellaneous

1. Dearborn Valve and Fitting  
PO Box 847  
Wauconda, IL 60084  
(630) 526-6800
2. Newark Electronics  
1365 Wiley Road  
Schaumburg, IL 60173  
(847) 310-8980
3. Revere Electric Supply  
617 Church Road  
Elgin, IL 60123  
(630) 741-8900
4. MAXTEK INC.  
2908 Oregon court  
Torrance, CA 90503  
(310) 320-6604
5. E Besler & Co.  
115 Martin Lane  
Elk Grove Village, IL 60007  
(630) 364-0300

# Opposed-Cathode System

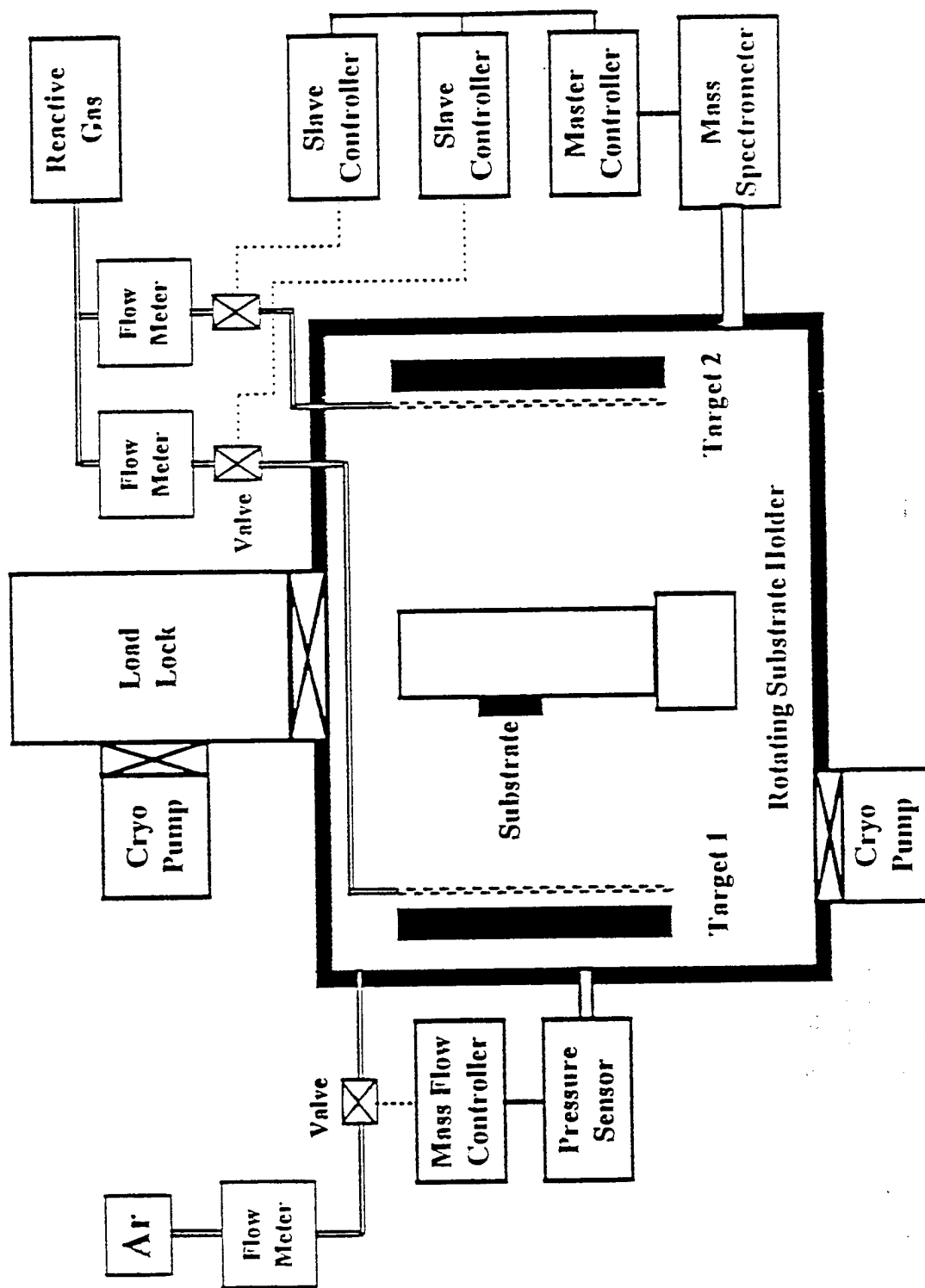


Figure 1. Schematic representation of the ceramic coating deposition system.